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Abstract

Agilent has developed the High Matrix Introduction (HMI) accessory for ICP-MS as an alternative to conventional dilution. The HMI modifies the sample introduction

tolerance to high-matrix samples. The Agilent High Matrix Introduction (HMI)¹ accessory reduces sample matrix load on the plasma, making it possible to directly measure sample solutions with TDS exceeding 1%. As a result, samples previously mea-

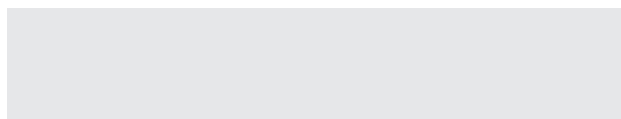


Table 2. Analytico 3-Sigma Required Detection Limits for Soils

Analyte	Soil dry wt (mg/kg)	After 50x dilution (µg/L)
	0.17	3.4
r	15	300
	5	100
	3	60
	13	260
	17	340
	0.05	1
A	4	80
A	1	20
̂	10	200
	1	20
	6	120
B	15	300
	1	20
	1.5	30
	1	20
̂	0.1	2
̂	10	200
	3	60

MDLs were calculated as 3-sigma of 10 replicates of a low-level (between one to three times the required MDL) spiked sand sample measured consecutively and also measured on 10 different days over a 30-day period² (Table 3). For all regulated elements, the calculated MDLs exceeded the Dutch regulatory requirements by nearly an order of magnitude or better. So with the HMI operating at maximum effective dilution, the system has ample sensitivity for the application.

Analysis of Certified Reference Materials

Two certified reference materials (BCR-144R Domestic Sewage Sludge, IRMM, Belgium, and

Table 3. Calculated Method Detection Limits (mg/kg) According to the Requirements of Dutch Regulation AS3000

(1) = 3σ $10 \hat{\rho}_c \hat{\rho}_t \hat{\rho}_t$
 (2) = 3σ $10 \hat{\rho}_c \hat{\rho}_t \hat{\rho}_t$ ¹

Analyte - isotope	ORS mode	MDL (1) (mg/kg)	MDL (2) (mg/kg)	Dutch required MDL (mg/kg)
̂ 9	̂	0.042	0.046	0.1
51	̂	0.255	0.481	1
r 52	̂	2.300	4.517	15
59	̂	0.147	0.348	1
60	̂	0.770	0.922	3
63	̂	0.502	1.303	5
66	̂	1.704	3.104	17
A 75	̂	0.549	1.079	4
̂ 78	2	0.832	2.041	10
̂ 78	̂	1.064	1.991	10
95	̂	0.195	0.413	1.5
A 107	̂	0.278	0.701	1
114	̂	0.058	0.066	0.17
118	̂	0.589	1.353	6
121	̂	0.333	0.401	1
̂ 125	̂	1.217	2.112	10
B 135	̂	3.041	6.227	15
201	̂	0.014	0.025	0.05
203	̂	0.285	0.546	3
208	̂	1.197	2.844	13

FeNeLab River Clay, FeNeLab, Netherlands) were analyzed in replicate as part of the validation procedure. The CRMs were prepared in the same manner as standard soil samples and measured on 10 different days during a 30-day time period. Table 4 shows the results of replicate (n = 10) analyses of both CRMs in mg/kg. Recoveries ranged from 87 to 108%, well within the regulatory requirement of 80 to 110%.

² $c_c \hat{\rho}_r 10 \hat{\rho}_t \hat{\rho}_r r t$

Analyte	ORS Mode	FeNeLab River Clay			BCR-144R Sewage Sludge		
		Measured mg/kg (ave, n = 10)	Certified mg/kg	Rec. % (ave)	Measured mg/kg (ave, n = 10)	Certified mg/kg	Rec. % (ave)

Determination of Precision and Accuracy at High and Low Concentrations

In addition to analysis of replicate CRMs, both low-level and high-level spiked samples were analyzed in replicate (n = 10; 10 different days during 30-day time period) in order to determine both accuracy and precision over a wide range of concentrations (Table 5).

Sample Analysis

In order to test the long-term robustness of the HMI-equipped system, a 23-hour sequence consist-

Productivity

The operation of the HMI accessory does not adversely affect productivity in any way, since the HMI conditions are constant throughout operation and do not require any additional execution or stabilization time. Therefore, a given method run with HMI would take the same amount of time per sample as one run without HMI. HMI is also fully compatible with Agilent's time-saving pre-emptive and intelligent rinse functions, which minimize time wasted during both sample uptake and rinse-out. Furthermore, since HMI permits the direct analysis of undiluted samples of many types, the extra time associated with either manual or autodilution is saved, which considerably shortens the total time (prep plus analysis) required per sample. The improved stability as a result of HMI use can also minimize the need for recalibrations and sample reruns, further reducing the average run-to-run time. In this work, the average run time for a sample in a 23-hour, 235-sample sequence of undiluted soil digestates was 5.9 minutes, including acquisition in both H₂ and He modes.

Conclusions

Based on Analytico's evaluation, when compared with conventional autodilution for high TDS, high acid digests of soil and sludge samples, Agilent's

HMI interface provided a number of significant advantages.

- Speed – HMI does not require liquid dilution of sample and stabilization of diluted sample. It also permits the use of Agilent's pre-emptive rinse function, which allows rinsing of the sample tubing to begin before acquisition has finished.
- Low maintenance – There is no tubing to replace and no moving parts to maintain.
- Simple – There are no critical timing issues or plumbing common to continuous flow autodilutors.
- Flexibility – Since hardware changes or reconfigurations are not required after installation of HMI, the system can be switched between conventional mode and HMI mode on the fly.

These advantages have allowed Analytico to use a single 7500cx ICP-MS fitted with HMI to replace several instruments required for the analysis of these sample types, including conventional ICP-MS, ICP-OES, and a dedicated mercury analyzer.

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